ENHANCED SPATIAL RESOLUTION OF MAGNETIC RESONANCE MEASUREMENT AND APPLICATION TO WHOLE CORE SHALE

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ABSTRACT

Low field Nuclear Magnetic Resonance (NMR) has been widely used in laboratories for fluids and small core plug analysis to provide more accurate petrophysical measurements and for calibration of borehole log data. One fundamental limiting factor of NMR application for whole core or well logging is that the spatial resolution is limited by the length of the *rf*-coil. In addition, the end effects of finite coil length can compromise the accuracy of NMR measurement when the core is longer than the coil. Pulsed field gradient (PFG) technology may be used for slice selection in the laboratory; however, PFG-based method does not work well for rocks with short relaxation time, for example shales and other tight rocks, because the NMR signal decays significantly during the application of PFGs.

Herein we propose a method that provides high spatial resolution NMR measurement using the response map of the *rf*-coil coupled with a data acquisition and processing protocol that step-wise moves the *rf*-coil or the studied subject with equal increments of the desired spatial resolution. The main advantages of this method are that it:

- Provides a measurement of fluid content and images with any desired spatial resolution that is not limited by the length of the *rf*-coil or antenna used in the measurement. This can be especially useful in laboratory applications for samples longer than the coil and in well-logging where the studied objects are always much longer than the tool's antenna.
- Eliminates end effects from the finite length of the *rf*-coil.
- Enable acquisition of high-spatial resolution NMR relaxation data for samples with short relaxation times.
- Delineates sample heterogeneity facilitating the upscaling of NMR-based petrophysical properties.

We applied this method to acquire high-spatial resolution NMR measurement on shale whole cores.

INTRODUCTION

Horizontal drilling and hydraulic fracturing has revolutionized hydrocarbon production from unconventional shale and other tight reservoirs; however, the hydrocarbon storage and transport mechanisms remain poorly understood for these reservoirs. This understanding is important for predicting and optimizing production.

Low field (2 MHz) NMR has proved to be a powerful technology for estimating reservoir fluid contents and characterizing fluid transport properties. It has been widely used in laboratories for fluids and small core plug analysis to provide more accurate petrophysical measurements and for calibration of field log data. However, it has rarely been used on whole core samples. This is probably because in conventional whole cores, fluids would largely be expelled while cores are extracted from downhole. This is not the case for cores taken from unconventional source rocks, with nD permeability where the native fluids (except for gas) largely remain in the rock. Thus, estimating fluid content and characterizing fluid-solid interaction using NMR should reveal important information about the shale reservoirs that may not be available by any other method.

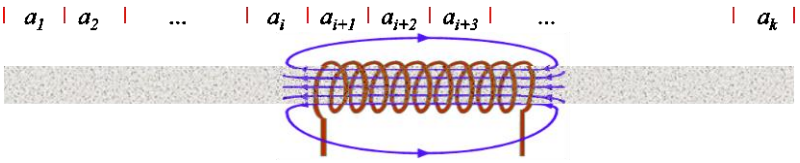

Fig. 1: Illustration of a long sample in a short *rf*-coil while the required resolution is smaller than the coil.

One fundamental limiting factor of NMR application for long whole cores or for well logging is that the spatial resolution is limited by the length of the radio-frequency (*rf*) coil. The length is a few inches for laboratory instrument and a few inches to a few feet for well-logging tools. In typical NMR measurements the length of sample is shorter than that of coil. In this case, the length of coil is assumed to be infinite, which ensures the *rf* field strength and measurement sensitivity across the sample is homogenous. For NMR measurements on long samples or essentially log applications where the sample is longer than the *rf*-coil, one must consider the end effect of a finite coil for accurate measurement. **Fig. 1** illustrates a long sample within the *rf* magnetic field generated by a solenoid coil. On top of the figure, the labeled signal along the core has a smaller spatial resolution than the coil length. **Fig. 1** shows two problems for a conventional NMR measurement: first, the best resolution that can be achieved in a simple experiment would be approximately the length of the *rf*-coil, which can be much larger than the required resolution. Second, the measurement is not accurate because the measured signal includes signal from the two ends of the coil which have much different detection sensitivity comparing to the homogeneous central part within the coil.

A solution to these problems to obtain high spatial resolution NMR measurement is to use PFG technology for slice selection, which is widely used in medical magnetic resonance imaging (MRI). However, high quality PFG can significantly increases the cost of the NMR instruments. In addition, it cannot be used to quantitatively analyze materials with short transverse relaxation times, T_2, because the signal decays to a very small value during the application of PFG. Many tight rocks, such as shales, tight sandstones, and tight carbonates, have T_2 components of 1 ms or less. For example, if a PFG with length 0.6 ms is used, after a pair of PFG the signal with 1 ms T_2 decays to exp(-1.2/1) = 0.3. In this case, 70% signal is lost and more will be lost if longer PFG is used or the T_2 is shorter.

In this paper, we describe a method that

- Provides NMR measurement with any desired spatial resolution that is not limited by the length of the *rf*-coil or antenna used in the measurement. This can be especially useful in laboratory applications for samples longer than the coil and in well-logging where the studied objects is always much longer than the tool antenna.
- Eliminates end effects from the finite length of the *rf*-coil.
- Enables acquisition of high spatial resolution NMR relaxation data for samples with short relaxations

The developed technique was used to log 35 whole shale cores (4 inches in diameter and a foot long) from two Middle East wells and to acquire T_2 spectra and fluid content with one inch spatial resolution along the cores using an NMR spectrometer with a four-inch long *rf-coil*.

METHOD

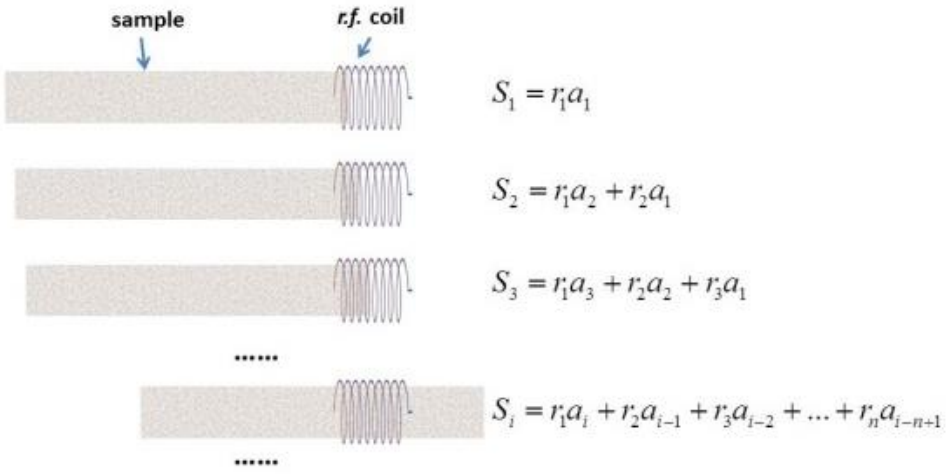

Fig. 2: Illustration of the data acquisition by step wisely moving the sample through the coil. The equations show the acquired signal at each step.

Mathematic description of general method The fluid content of the whole core can be expressed as $\{a_1, a_2, a_3, \ldots, a_k\}$, as showed in **Fig. 1**. The total length of the core is then

$k \times l$ where l is the spatial resolution and k is the number of sections the sample has. Our method requires a response map of the *rf*-coil with spatial resolution equal to or a fraction of the desired resolution l. This can be experimentally measured or calculated if the shape of the coil is known.

The method also needs to acquire the NMR data for the long coil stepwise with steps equal to or less than the spatial resolution l. The stepwise data acquisition can be achieved in two ways: (1), moving a sample though the *rf*-coil. This is normally done in the laboratory when the sample is longer than the *rf*-coil. **Fig. 2** shows how this stepwise passing of sample through a coil. (2), moving the *rf*-coil along the measurement subject. This is a typical data acquisition procedure in logging where the logging tool moves along the wellbore from bottom to top.

For both data acquisition methods, the detected signal at step i can be expressed as:

$$S_i = r_1 a_i + r_2 a_{i-1} + r_3 a_{i-2} + r_4 a_{i-3} + \cdots + r_n a_{i-n+1} \quad (1)$$

$\{r_1, r_2, r_3, \ldots, r_n\}$ represents the one dimensional response map of the *rf*-coil. It is convenient to express Eq. (1) in matrix form as

$$\boldsymbol{S} = \boldsymbol{R}\boldsymbol{A} \quad (2)$$

with

$$\boldsymbol{S} = \begin{bmatrix} s_1 \\ s_2 \\ \vdots \\ s_{k+n-1} \end{bmatrix} \quad (3)$$

$$\boldsymbol{R} = \begin{bmatrix} r_1 & & & & & & & & & \\ r_2 & r_1 & & & & & & & & \\ r_3 & r_2 & r_1 & & & & & & & \\ & \ddots & & & & & & & & \\ & & r_n & \cdots & r_3 & r_2 & r_1 & & & \\ & & & & & & & \ddots & & \\ & & & & & & & & r_n & r_{n-1} & r_{n-2} \\ & 0 & & & & & & & r_n & r_{n-1} \\ & & & & & & & & & r_n \end{bmatrix} \quad (4)$$

$$\boldsymbol{A} = \begin{bmatrix} a_1 \\ a_2 \\ \vdots \\ \\ \\ \\ \\ \\ a_k \end{bmatrix} \tag{5}$$

Matrix $\boldsymbol{S}$ is the measured NMR signal from the spectrometer which contains coil end effects, and, thus, has a spatial resolution slightly larger than the length of the *rf*-coil. Matrix $\boldsymbol{R}$ is the response matrix and is determined by *rf*-coil and also the way the NMR data acquisition was carried out. Matrix $\boldsymbol{A}$ is the desired NMR signal along the sample with high spatial resolution.

$\boldsymbol{A}$ is obtained by solving Eq. (2). Note that the dimension of $\boldsymbol{S}$, $\boldsymbol{R}$, and $\boldsymbol{A}$ are $(k+n-1) \times 1$, $(k+n) \times k$, and $k \times 1$, respectively; therefore, obtaining $\boldsymbol{A}$ is an over-determined problem because the dimension of $\boldsymbol{S}$ is larger than $\boldsymbol{A}$.

To solve Eq. (2), we convert Eq. (2) to:

$$\boldsymbol{R}'\boldsymbol{S} = (\boldsymbol{R}'\boldsymbol{R})\boldsymbol{A} \tag{6}$$

where $\boldsymbol{R}'$ is the transpose of matrix $\boldsymbol{R}$. In Eq. (6), $\boldsymbol{R}'\boldsymbol{R}$ is a $k \times k$ matrix and $\boldsymbol{R}'\boldsymbol{S}$ is a $k \times 1$ matrix. Then:

$$\boldsymbol{A} = (\boldsymbol{R}'\boldsymbol{R})^{-1}\boldsymbol{R}'\boldsymbol{S} \tag{7}$$

where $(\boldsymbol{R}'\boldsymbol{R})^{-1}$ is the inverse of $\boldsymbol{R}'\boldsymbol{R}$. All the terms on the right of Eq. (7) are know: matrix $\boldsymbol{S}$ is the measured NMR signal using the stepwise approach and matrix $\boldsymbol{R}$ represents the response function constructed as Eq. (4) using the *rf*-coil response map. Thus, evaluating the right side of Eq. (7) gives us $\boldsymbol{A}$**:** the NMR signal with spatial resolution predetermined as *l*.

For a typical CPMG echo train data, each acquired signal, S_i, includes *m* echoes. Both $\boldsymbol{S}$ and $\boldsymbol{A}$ are matrixes, which can be expressed as;

$$\boldsymbol{S} = \begin{bmatrix} s_{1,1} & \cdots & s_{1,m} \\ \vdots & \ddots & \vdots \\ s_{k+n-1,1} & \cdots & s_{k+n-1,m} \end{bmatrix} \tag{8}$$

$$\boldsymbol{A} = \begin{bmatrix} a_{1,1} & \cdots & a_{1,m} \\ \vdots & \ddots & \vdots \\ a_{k,1} & \cdots & a_{k,m} \end{bmatrix} \tag{9}$$

Each row in ***A*** represents the echo train with full intensity and free of *rf*-coil end effects. Inversion of each echo train in ***A*** produce NMR T_2 spectra along the long sample.

Computation programs were coded with Matlab to solve Eq. (7) using a general linear least squares method for singular value decomposition. Inversion program of the echo trains in ***A*** was also coded with Matlab to obtain the NMR T_2 spectra.

NMR measurement An NMR spectrometer from Ecotek Corp was used for data acquisition. It operates at 1.83 MHz with probe diameter 108 mm allowing the 4-inch full-diameter cores to be pushed through as illustrated in **Fig. 3**. The *rf*-coil located in the middle of the magnet was approximately 4 inches long, and, thus, could not 'see' the whole one foot long whole core in one single measurement.

The NMR signal was acquired using CPMG [1, 2] pulse sequence. The echo time, *TE*, was 0.17 ms (milliseconds) and inter-scan delay was 500 ms.

Fig. 3: Illustration of a whole core in Teflon shrink-wrap in the spectrometer probe. The large gray box encloses the magnet and *rf*-coil. A separate box on the left contains the control console and the *rf* amplifier.

A series of data acquisitions were made, beginning with the top of the core flush against the start of the sensitive window of the *rf* probe. This way, the initial scan should always be a noise signal. NMR acquisitions are continued along the length of the core by pushing it manually through the 4-inch probe, one inch at a time. The acquisition continued until the core completely passes through the sensitive window, once again ending in a pure noise signal. For a foot long core to pass through the 4-inch probe, it required 17 acquisitions.

To measure the response map of the *rf*-coil with resolution equal to 1 inch, a disk-shaped polycarbonate standard was prepared. The inner diameter and thickness were approximately 3.68 and 0.94 inches, respectively. It was filled with double-distilled water doped with $CuSO_4$ with a concentration of approximately 215 ppm to shorten the water T_2 to 70 ms. This standard contained 178.2 g of water (approximately 178.2 ml) at room condition. To map out the *rf*-coil response, the standard sample was place into the coil and manually pushed through the magnet one inch at a time. The NMR CPMG echo was acquired at each step.

The standard sample also served to calibrate the fluid content in the whole core samples. The percent fluid content in the rock is calculated as:

$$p_{Fluid} = r\frac{M_0^{core}/V^{core}}{M_0^{w}/V^{w}} \times 100\% \qquad (10)$$

where r is the measured response factor at the position in the coil, M_0^w and V^w are the measured NMR signal and volume for the standard water sample, and M_0^{core} and V^{core} are the measured NMR signal and volume for the core sample.

RESULTS AND DISCUSSION

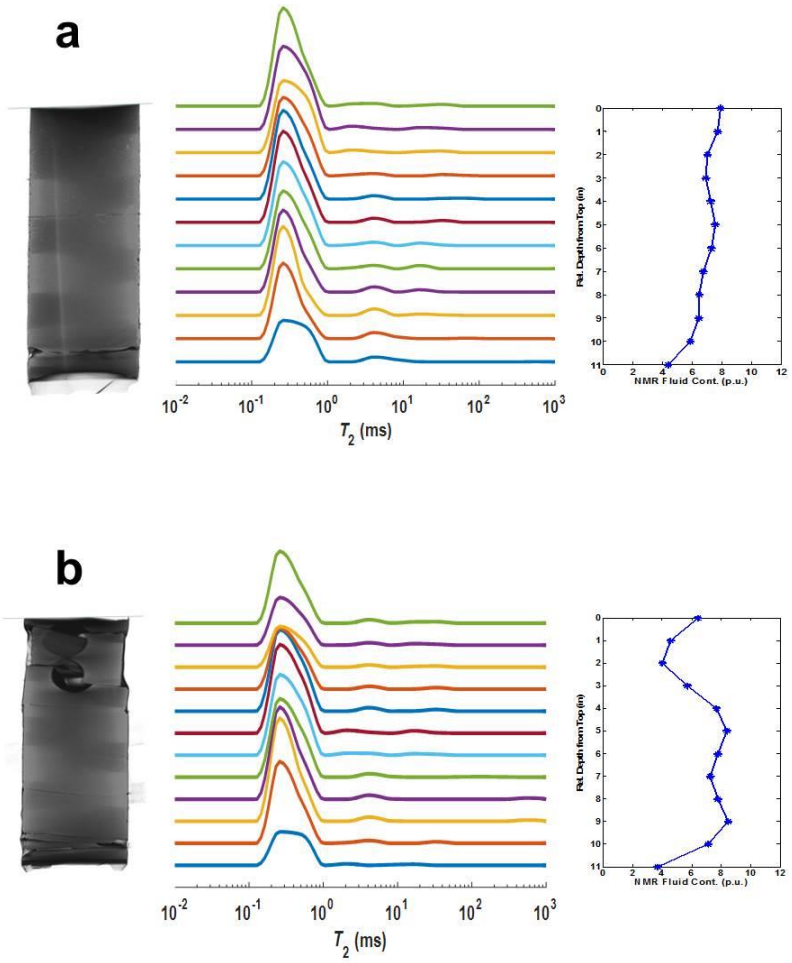

Fig. 4: CT image, NMR measured fluid content and NMR T_2 spectrum of every inch along two one-foot long whole cores (a) and (b) measured with the 4 inch long *rf*-coil. From left to right for the two samples are: slice of CT image, T_2 spectra, and fluid content from the T_2 measurement.

Response map of rf-coil The response map of the *rf*-coil represents the detection sensitivity at each position for the coil. It is determined by both the excitation of the nuclear spins by

the *rf* pulse from the coil and detection of the current generated by the excited nuclear spin. Table 1 lists the acquired relative signal intensity at each relative position inside the probe measured using the standard sample for the NMR spectrometer used in this test. The relative signal intensity provides the coil response curve. The data shows that the coil response is quite uniform in the middle of the coil and falls off quickly at the two ends of the coil. It can be seen that fluids at positions 1 and 5 contribute less than half of the full signal. At positions 0 and 6 fluids contribute very little to the detected signal and will be neglected hereafter. This response map is unique and can be varied for different spectrometers and different coils for the same spectrometer.

Table 1 - Acquired NMR signal intensity of the standard sample in the coil

Relative Position	0	1	2	3	4	5	6
Relative Signal Intensity *r*	0.01	0.35	1.00	0.98	0.98	0.49	0.05

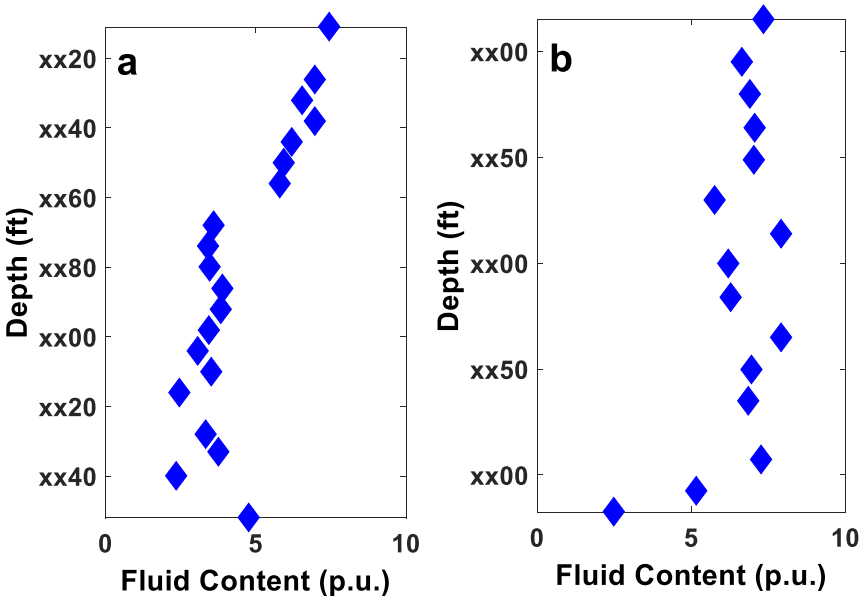

Fig. 5: NMR measured fluid contents for the 35 whole cores from two wells acquired at different depth.

High spatial resolution NMR Fig. 4 shows representative NMR results for two whole cores (a) and (b) using the stepwisely acquired data. On the left is the slice images taken from the whole core CT, showing that the whole core broke off at several places; in addition, two plugs were drilled before our measurements from the core (b). The middle column is the T_2 spectrum for each inch of the whole core and the last column shows the summation of the T_2 spectrum which is the total fluid content for each inch of the core. The variation of the NMR measured fluid content in the right column over the one foot long core is consistent with the integrity of the cores which is more obvious for sample (b). This strongly suggests that the NMR result of one inch spatial resolution is quite accurate. As a result, we have achieved 1 inch spatial resolution from 12 inch length whole core sample using a 4 inches long *rf*-coil by the proposed method.

The NMR T_2 spectra show that the main T_2 peak are largely at the same value, indicating the pore system over each inch of the sample are relatively similar through whole core sample. It is also interesting to note that the NMR T_2 peak is 'smeared' at the locations where the whole core is not intact.

Fig. 5 shows the NMR measured fluid content for the 35 whole cores from two wells (a) and (b). Each point in the plot represents the average fluid content from 12 NMR spectra over the 1foot long whole core. The results suggest that in the studied part well (a) has two distinct zones separated at XX60 ft while well (b) might have some layered structure in the middle of the studied zone. On well (b), there could be some large changes at the bottom of the studied zone in the formation.

All the cores studied here were from a shale gas reservoir and have been kept in the laboratory for more than a year. The original gas should have bled off and the NMR measured fluid in the cores can only be water. The data shows the water content is quite high, approximately 8 p.u. in the top zone of well (a) and most of the zone in well (b).

CONCLUSION

A method has been developed that can acquire NMR data with spatial resolution higher than the length of the *rf*-coil which has usually been considered as the limit of the spatial resolution without using gradients. In addition, this method can acquire accurate data by eliminating the end effects of the *rf*-coil. More importantly, it only uses hard pulses and does not require the application of gradient pulses, which enables the acquisition of NMR data on samples with very short relaxation times. We have successfully demonstrated the current method on shale whole cores to acquire NMR T_2 spectra with 1inch spatial resolution using a 4 inches *rf*-coil. The NMR whole core logging method in this work can be used as a non-destructive tool to estimate fluid content in whole cores at well-sites or at core handling facilities.

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REFERENCE

[1] Carr HY, Purcell EM. Effects of Diffusion on Free Precession in Nuclear Magnetic Resonance Experiments. *Physical Review*. 1954;94:630-8.
[2] Meiboom S, Gill D. Modified Spin-Echo Method for Measuring Nuclear Relaxation Times. *Review of Scientific Instruments*. 1958;29:688-91.